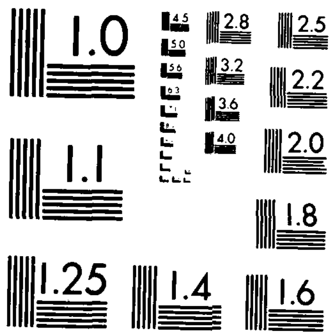
AD-A189 691 SUBMICROSCOPIC DEFORMATION IN CEMENT PASTE AND MORTAR AT HIGH LOAD RATES(U) KANSAS UNIV LAWRENCE DEPT OF CIVIL ENGINEERING D DARWIN 23 OCT 87 AFOSR-TR-87-1733 1/1

UNCLASSIFIED AFOSR-86-0207 F/G 11/2 NL



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

SUBMICROSCOPIC DEFORMATION IN CEMENT PASTE AND MORTAR AT HIGH LOAD RATES

DOD-University Research Instrumentation Program

Scanning Electron Microscope and Energy Dispersive X-Ray Analysis System

David Darwin

University of Kansas

Department of Civil Engineering

Lawrence, KS 66045

23 October 1987

Final Report for Period 1 August 1986-31 July 1987

Prepared for:

Air Force Office of Scientific Research

Bolling Air Force Base

Washington, DC 20332

GRANT NO. AFOSR-86-0207

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION Unclassified	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited.
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S) AFOSR-TR- 87-1733
6a. NAME OF PERFORMING ORGANIZATION University of Kansas 6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION Air Force Office of Scientific Research
6c. ADDRESS (City, State, and ZIP Code) Lawrence, Kansas 66045	7b. ADDRESS (City, State, and ZIP Code) Bolling Air Force Base Washington, DC 20332
8a. NAME OF FUNDING/SPONSORING ORGANIZATION AFOSR 8b. OFFICE SYMBOL (If applicable) NA	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER AFOSR-86-0207
8c. ADDRESS (City, State, and ZIP Code) Bolling AFB, DC 20332-6448	10. SOURCE OF FUNDING NUMBERS PROGRAM ELEMENT NO. 61102F PROJECT NO. 2917 TASK NO A1 WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification)
Submicroscopic Deformation in Cement Paste and Mortar at High Load Rates

12. PERSONAL AUTHOR(S)
Darwin, David

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Final	FROM 860801 TO 870731	871023	15

16. SUPPLEMENTARY NOTATION

17. COSATI CODES		
FIELD	GROUP	SUB-GROUP

18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
Cement, concrete, electron microscopes, image analysis, microcracks, microscopic, mortar, strain rate, submicrocracks, x-ray analysis.

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

Research into the submicroscopic behavior of the cement paste and mortar constituents of concrete is enhanced through the acquisition of an integrated scanning electron microscope, energy dispersive spectrometer, image analysis system. The combined system is used to obtain crack surveys of cement paste and mortar loaded in uniaxial compression at strain rates ranging from 0.3 to 300,000 microstrain per second. Research background, equipment selection procedures, equipment capabilities, installation, and on-site evaluation are described. Crack dimensions, orientations, and locations are determined using the combined instrumentation. The surface crack distributions are converted to three-dimensional crack distributions, which are in turn used to estimate the portion of the total deformation due to cracking. The new instrumentation provides a major improvement in both the quality and the quantity of crack data available for analysis.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT ☐ DTIC USERS	21. ABSTRACT SECURITY CLASSIFICATION Unclassified
22a. NAME OF RESPONSIBLE INDIVIDUAL Spencer T Wu	22b. TELEPHONE (Include Area Code) (202) 767-6962 22c. OFFICE SYMBOL AFOSR/NA

DD Form 1473, JUN 86 Previous editions are obsolete. SECURITY CLASSIFICATION OF THIS PAGE

UNCLASSIFIED

SUBMICROSCOPIC DEFORMATION IN CEMENT PASTE AND MORTAR AT HIGH LOAD RATES

DOD-UNIVERSITY RESEARCH INSTRUMENTATION PROGRAM SCANNING ELECTRON MICROSCOPE AND ENERGY DISPERSIVE X-RAY ANALYSIS SYSTEM

INTRODUCTION

Research into the submicroscopic behavior of cement paste and mortar has been underway at the University of Kansas since 1980. Since 1985, under Air Force Office of Scientific Research support, emphasis has been placed on submicroscopic deformation at high strain rates.

The aim of the research is to determine the structural changes that occur in the cement paste and mortar constituents of concrete at high rates of loading and to correlate these changes with observed material behavior, based on the applied strain and strain rate. Major emphasis has been placed on submicroscopic cracking.

Specimens of cement paste and mortar are subjected to uniaxial compression at strain rates ranging from 3.0×10^{-7} to 3.0×10^{-1} sec^{-1}. Both dry and saturated specimens have been evaluated. Crack dimensions, orientations, and submicroscopic structures through which the cracks pass are determined using a scanning electron microscope (SEM). The surface crack distributions obtained with the SEM are converted to three-dimensional crack distributions. The three-dimensional distributions are used in turn to estimate the portion of the total deformation due to cracking.

The research is designed to shed light on the mechanisms that control the behavior of concrete and its constituent materials and to provide a rational basis for developing descriptive material laws, much in the same way that metal plasticity laws are firmly anchored in dislocation slip theory. With this knowledge, analysts will be in a position to formulate mathematical behavior laws based on the structural changes that actually take place in the concrete and will no longer be limited to empirical expressions.

Prior to the acquisition of the equipment described in this report, crack studies of cement paste and mortar were limited by the tedious manual procedures used to measure and categorize the cracks. Only a portion of each specimen could be studied, and careful attention was necessary to insure that the crack surveys remained objective.

To help alleviate these drawbacks, a proposal was submitted under the DOD-University Research Instrumentation Program for a scanning electron microscope and energy dispersive spectrometer system. This grant was funded by the Air Force Office of Scientific Research and later combined with additional funding from AFOSR to include a fully integrated image analysis system.

This report presents the research background and justification for the equipment, a description of the search and evaluation procedures used to select the instrumentation, a summary of the capabilities of the equipment that was selected, and a description of the installation and on-site evaluation of the systems.

BACKGROUND

The main experimental emphasis of the current study is to catalog the submicroscopic structural changes that occur in cement paste and mortar at high rates of loading in compression. To obtain this information, specimens are loaded to the desired strain and then unloaded. Slices of the material, 3 mm thick, are removed from the test specimens and prepared for viewing in a scanning electron microscope. The purpose of the preparation procedures is to insure that no evaporatable water will escape from the specimen while it is under vacuum in the SEM. Virtually all research on cement paste, including work at the University of Kansas prior to the current grant, has used oven drying to remove the water. The dried specimens are coated with a conducting material, usually gold-palladium, before viewing in the microscope.

In the original survey technique, structures in cement paste were cataloged based on well known morphologies. Crack measurements were made manually in the "TV" scanning mode over 10 evenly spaced strips across the 3 mm width. The procedure was quite time consuming, and a typical specimen took about 2½ hours. To help insure objectivity, crack surveys were limited to a single specimen per day. The operator carried out the surveys at the same time on each day, and a double blind marking procedure was used to insure that the operator did not know which specimens were being surveyed. Following these procedures, individual operators were able to develop consistent criteria for crack identification. However, limiting the differences between operators was not an easy task.

Although the techniques were not ideal, a great deal of useful data was obtained using these procedures. However, a number of other drawbacks impacted the study. First, drying of the specimens introduced additional cracks. An analytical procedure was used to account for the "preparation" cracks; however, another procedure has the potential to significantly reduce the number of specimen preparation cracks and thus leave the load-induced cracks undisturbed.

Rather than drying the specimens, the new procedure quick freezes the specimens to -210°C using nitrogen slush. This drops the temperature of the material so rapidly that ice crystals do not form, and the water within the specimen retains its amorphous structure. The specimens are then viewed within the SEM using a cold stage to maintain the low temperature.

When this technique is used, the crack density for nonloaded specimens is lower than obtained with dried specimens, indicating that preparation by quick freezing is less traumatic than drying. However, the structures commonly seen on dry SEM specimens are not visible on frozen specimens. In their place, new structures appear. To evaluate these structures requires an energy dispersive x-ray analysis or energy dispersive spectrometer (EDS) system. This system was not available at the University of Kansas.

An EDS system is used in conjunction with an SEM and could have been added to the existing University SEM. However, that microscope was nearly nine years old and no longer represented the state-of-the-art. It placed some restrictions on the study, and its limitations would have prevented full advantage to be taken of an x-ray analysis system. For that reason, a new scanning electron microscope was also proposed with features designed to enhance the current study.

The proposed microscope provided a number of advantages over the existing microscope, since it included a motorized stage that could be controlled by the computer within the EDS system and also contained an automatic focus and automatic stigmator unit, which could reduce the $2\frac{1}{2}$ hour analysis time by 50 to 60 percent, and greatly reduce problems with operator fatigue.

The proposed microscope also included a backscattered electron detector, which allows direct observation of material surfaces without the application of a gold-palladium coating and provides much clearer images of voids and cracks than does the normally used secondary electron detector.

Following funding for the instrumentation and during evaluation of available equipment, a proposal for additional funding to add an image analysis system was sent to AFOSR. This equipment has the additional capabilities of allowing fully automated crack surveys to be carried out, and greatly increasing both the accuracy of the surveys and the extent of the material that can be surveyed. This proposal was also funded, allowing the scanning electron microscope, x-ray analysis system, and image analysis system to be integrated prior to delivery to the University of Kansas.

The following section provides a description of the evaluation and selection process for all three portions of the final instrumentation systems.

EQUIPMENT EVALUATION AND SELECTION

Equipment evaluation and selection consisted of three phases: (1) study of manufacturers' literature and discussions with manufacturers' representatives, (2) on-site visits to observe the equipment, and (3) comparisons of the instrumentation based on the analysis of standard specimens.

In all three phases, a series of criteria were used to evaluate the equipment. For the scanning electron microscope, the criteria included the quality of the monitor image; the quality of the micrographs; the ease of identifying constituents, cracks, and voids within cement based materials; clarity of both secondary electron and backscattered electron images; resolution of images at high magnifications; the availability of a TV-rate backscatter detector, autofocus and autostigmator units, and a computer interface; and the ease of operation. The criteria used for the energy dispersive x-ray analysis system included the accuracy of the elemental analysis; ease of operation of the instrumentation; and the user-friendly nature of the software. The criteria for selection of the image analysis system included the speed of image acquisition; the efficiency of image averaging; the sophistication of the image acquisition and analysis software; the ability of the system to interface with the microscope and the x-ray analysis system; the ability to control the stage and autofocus capabilities of the microscope; and the ability for long-term, fully automated analysis of specimens. This last factor is especially important for carrying out crack surveys of cement paste and mortar.

A study of the manufacturers' literature and discussions with manufacturers' representatives began in May 1986 and included a preliminary evaluation of seven scanning electron microscopes (Amray, Cambridge, Gatan, Hitachi, ISI, JEOL, and Philips), four x-ray analysis systems (EDAX, KEVEX, Princeton Gamma-Tech, and Tracor Northern), and five image analysis systems, (EDAX, KEVEX, LeMont, Princeton Gamma-Tech, and Tracor Northern). Based on an analysis of the literature and discussions with individual representatives, on-site visits were arranged for three scanning electron microscopes, three x-ray analysis systems, and four image analysis systems. Three of the image analysis systems were integrated with EDS systems. One EDS manufacturer (Tracor Northern) also produced a separate, stand-alone image analysis system. The fourth supplier, LeMont Scientific, produced an image analysis system which was separate, but designed to be integrated with both EDS and SEM systems.

The on-site visits took place during the period of August through November 1986. All visits were to a company's applications laboratory, with the exception of the Princeton Gamma-Tech x-ray analysis system, which was viewed in a commercial laboratory in Dallas, Texas.

Scanning Electron Microscope

The scanning electron microscopes evaluated in detail were the Hitachi 570, the JEOL 840, and the Philips 515. Each microscope represented the top-of-the- line for standard scanning electron microscopes produced by each of the companies. The on-site visits indicated that the Philips 515 had superior operational characteristics. It produced superior monitor images and had excellent resolution at both low and high magnifications over the full range of accelerating voltages, used at an excellent TV rate backscattered electron detector, had the best automatic focus and automatic stigmator unit, and was the only unit to have a computer interface.

To further evaluate the instruments, standard specimens of cement paste and mortar were photographed over a range of accelerating voltages and magnifications using both secondary and backscattered electron detectors. An evaluation of the micrographs indicated that, in all cases, the micrographs produced by the JEOL 840 and Philips 515 exceeded in quality those produced the Hitachi 570. The Philips micrographs were superior in all cases for secondary electron detector images, with a special advantage at low voltages. The JEOL micrographs were slightly better than the Philips micrographs using the backscattered electron detector. However, the backscattered electron detector produced by JEOL was not available as a TV rate instrument. A Philips 515 scanning electron microscope was selected as the best instrument.

Energy Dispersive X-Ray Analysis System

Three x-ray analysis systems were evaluated on-site. These included the EDAX 9900, the Princeton Gamma-Tech System 4, and the Tracor Northern TN-524 systems. The EDAX 9900 possessed the greatest ease of operation, as well as the most user-friendly software for basic elemental analysis. It also had the best display of x-ray spectra. Especially attractive about the EDAX system was the operator's console, which includes an interactive display and dynamic function keys. The on-site visits included or were followed up by the analysis of a reference standard to determine the accuracy of the standard-less quantitative analysis capabilities of the systems. Analysis was carried out on the EDAX 9900 and Tracor Northern TN-524 systems, but not on the Princeton Gamma-Tech System 4, because of the low evaluation that that system received during the on-site visit. In its place, an analysis was carried out on a KEVEX 8000 EDS system. A Tousimis glass reference standard of known elemental composition was utilized for the evaluation. The quantitative analyses performed (see Table 1) showed the EDAX 9900 to be the clear choice. In the final evaluation, the EDAX system proved to be not only the most effective elemental analysis system but also the most cost-effective system available on the market.

Image Analysis System

During the analysis of the EDS analysis systems, the built-in image analysis systems were studied in addition to the stand alone Tracor Northern TN-8500 image analysis system. One additional site visit, to view the LeMont OASYS image analysis system, was made to evaluate that system and its ability to interface with both SEM and EDS systems. An evaluation of the systems indicated that the LeMont OASYS was far ahead in terms of sophistication of the software, speed of image acquisition, efficiency of image averaging, and ability to interface with both SEM and x-ray analysis systems. The LeMont system also had the ability to control a motorized microscope stage, contained its own autofocus system, and was the only system with a long-term fully-automated analysis capability. The LeMont OASYS system was selected as the best instrument.

EQUIPMENT CAPABILITIES

The overall system consists of a Philips SEM 515 scanning electron microscope, an EDAX 9900 energy dispersive spectrometer, and a LeMont OASYS image analysis system.

The Philips 515, in addition to the standard secondary electron detector, has a true TV rate quad backscattered electron/multifunction detector, a motorized stage, a lanthanum hexaboride (LaB_6) gun system to increase gun brightness, a computer interface for external control, and a motorized stage. The system has special capabilities for operating at low voltage, and with a multifunction detector can provide cathodoluminescence images, as well as backscattered electron images. The system also includes an autofocus/autostigmator unit. The SEM is especially easy to operate.

The EDAX 9900 energy dispersive spectrometer includes an ECON IV detecting unit that can operate both with a beryllium window and in a windowless mode for detection of elements down to boron (at. no. 5). The system is fully integrated with the Philips 515 and provides for full standard based and standardless quantitative analysis. The system provides for selectable energy ranges, a definable preset based on live time, clock time, or peak count, automatic energy calibration, two memories for comparison, autoscaling for vertical full-scale, multiple window setting selection for x-ray mapping, and x-ray take-off angle and specimen tilt calculation.

The LeMont OASYS image analysis system is fully integrated with the Philips 515 and EDAX 9900. The LeMont system provides full automation for specimen scanning, image acquisition, and image analysis. The system provides X-Y stage control and automatic image focusing. The system is also capable of controlling parameters such as magnification, spot size, and scan speed. Specimens can be analyzed in real time, including elemental analysis, or images can be stored for additional enhancement and analysis. The system enhances images through procedures such as edge detection, inversion, filtering, erosion, dilation, and line profile. Features can be measured and classified based on single measurements or combinations of parameters, such as gray level, size, shape, width, length, aspect ratio, orientation, perimeter, and elemental composition.

INSTALLATION AND EVALUATION

The Philips and the EDAX systems were delivered to LeMont Scientific in State College, Pennsylvania, early in May 1987. LeMont completed its interfacing and delivered the systems to the University of Kansas in August 1987. The systems were installed and calibrated during August, September, and October 1987. System evaluation for acceptance included over 50 hours of operation during the months of September and October 1987. Users manuals were followed, images obtained, micrographs taken. The guaranteed resolution of the microscope was demonstrated. A number of standard specimens, including aluminum, copper, and silicon were analyzed using the energy dispersive spectrometer, and samples of paste, mortar, sludge, and air particulates were studied using the microscope, EDS, and image analysis systems. During this period, a number of weaknesses in the system were uncovered. These included improper operation of one of three vacuum pumps on the microscope, sensitivity of the EDAX system to variations in power, and a lack of accuracy in the magnification readings provided by the LeMont image analysis system when operating in the autofocus mode. The first two problems have been corrected, the third shortcoming will be corrected in the near future and does not prevent operation, since the correct magnification can be obtained directly from the Philips microscope.

Overall, images provided by the system are clearly superior to those provided by the older system on campus. Work is now underway to put the new system capabilities to work to evaluate specimens of cement paste and mortar subjected to a range of compressive strains and strain rates.

SUMMARY

The need to obtain high quality data on load-induced cracks in cement paste and mortar led to the development of proposals for a scanning electron microscope, energy dispersive spectrometer, and image analysis system for the University of Kansas. A detailed evaluation process was used to select instruments that will meet the needs of the current study. The equipment provides for full quantitative analysis and includes a backscattered electron detector with true TV rate imaging, a motor driven stage for accurate traverse control, and an autofocus/autostigmator unit for rapid operation. The image analysis system will allow fully automated specimen scanning, image acquisition, and image analysis.

The system will allow cracks to be measured based on consistent, objective criteria and will not be affected by operator fatigue, a major concern prior to the acquisition of this equipment. Data acquisition will be far more accurate than obtained with manual procedures, and as a result, the entire surface of a specimen can be scanned in less than one-third the time it currently takes to survey 10 percent of the specimen area.

The system capabilities will allow additional information, such as distribution of pores, and selected elemental analysis to be obtained during the crack surveys. The system is configured to take full advantage of the capabilities of the scanning electron microscope and energy dispersive spectrometer systems and will make a major contribution to ongoing AFOSR-sponsored research at the University of Kansas.

Table 1-Comparison of Analyses by Energy Dispersive X-Ray Analysis Systems (Windowless or Light Element Mode) of a Tousimis Reference Standard

ELEMENTAL PERCENTAGES

Element	Analysis	EDAX	KEVEX	Tracor Northern
Si	33.95	34.63	34.65	34.39
Ti	0.21	0.17	0.05	0
Al	0.59	0.51	0.69	1.63
Fe	0.03	0	0.39	0
Ca	4.68	3.97	3.43	4.83
Mg	1.66	1.70	1.16	1.11
Na	11.60	11.60	12.39	10.76
K	0.35	0.38	0.22	0.29
S	0.10	0.08	0.09	0
O	46.83	46.96	46.93	47.00

END
DATE
3-88
DTIC